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RECLAMATION OF WASTE OIL

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(71) Applicant
BUSS AG;

(72) Inventor
FELIX WALTER HAURI

(74) Attorney or Agent
SPRUSON & FERGUSON

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71431/81 545950 C10M 11/00
63886/80 543178 C10M 11/00, C08G 65/32, 65/48

(57) Claim

1. An improved method of reclaiming waste mineral oil by alkali treatment, removal of water and of highly volatile components, separation of a tar fraction and distillation of the purified oil, wherein the improvement comprises, in combination, the steps of:

treating the waste oil with an alkali compound selected from alkali metal compounds and alkaline earth compounds, after the removal of water and highly volatile components and before separation of the tar fraction, and separating the tar fraction during distillation of the oil.

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1 METHOD AND APPARATUS FOR THE RECLAMATION OF WASTE OIL

2 A B S T R A C T

3 Waste oil is reclaimed by alkali treatment, removal
4 of water and of highly volatile components; separation
5 of a tar fraction and distillation of the purified oil.
6 In particular, the waste oil is treated with alkali only
7 after the removal of water and highly volatile components
8 and before separation of the tar fraction, and the tar
9 fraction is separated during distillation of the oil.
10 Apparatus for fractioning oil and oil-like materials,
11 particularly waste oil, by means of an evaporator and
12 a condenser for oil distillation, include in combination
13 at least two condensers arranged in series.

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1 METHOD AND APPARATUS FOR THE RECLAMATION OF WASTE OIL

2 BACKGROUND OF THE INVENTION

3 1. Field of the Invention

4 The subject invention relates to methods of reclam-
5 ation of waste or used oil and to apparatus for the
6 fractioning of oil and oil-like materials, particularly
7 waste oil.

8 2. Information Disclosure Statement

9 In view of economic and ecological concerns, the re-
10 clamation of waste oil, also referred to as used oil, and
11 in particular of used lubricating oil, is of ever increas-
12 ing importance. Accordingly, many and greats efforts
13 have already been made in order to develop satisfactory
14 methods for the reclamation of used oil. Such a method
15 must satisfy the demands of ecological and economical
16 concerns and the quality of the reclaimed oil as well.
17 Solids which are forming a stable suspension with the used
18 oil must be separated and the solved contaminations
19 removed.

20 The known methods always contain several distillation
21 steps, which are always associated with additional pro-
22 cessing steps, such as addition of acids, coagulation,
23 adsorption and filtration and possibly processing by
24 means of metallic sodium or sodium hydride for the purpose
25 of de-halogenation until purified oil is obtained. These
26 known processes require extensive apparatuses and are
27 very energy consumptive. Also, they either produce
28 voluminous and ecologically unsound residuals or require
29 utilization of highly reactive chemicals requiring special
30 safety procedures.

1 In order to avoid the utilization of highly reactive
2 chemicals and in order to avoid the development of
3 voluminous residuals, US Patent 4,333,822 by A. Tkac et
4 al., issued June 8, 1982, and herewith incorporated by
5 reference herein, proposes a method for yielding purified
6 oil, wherein the waste oil is passed through a sedi-
7 mentation pool where coarse contaminations and a part of
8 the water present are separated, is treated with alkali
9 solution or lye at a temperature of 120 to 150° C in order
10 to remove acidic oil components and additive residuals,
11 is for removal of further water and volatile components
12 subjected to a first thin-layer distillation, and for
13 destruction of the suspension to a second thin-layer
14 distillation. The resulting distillate, which still
15 contains entrained solid suspended parts, is fed to a
16 third thin-layer distillation for effecting the actual
17 re-refining process for reclamation of the cleansed oil.
18 In order to render a purified product, this known method
19 requires an additional distillation step after the
20 suspension has been destroyed by means of distillation.
21 This is very expensive as far as required equipment and
22 energy consumption are concerned. Furthermore, the oil
23 thus yielded does not completely satisfy quality require-
24 ments as to purity and particularly with regard to odor
25 and color, whereby the application of that known method
26 is limited.

SUMMARY OF THE INVENTION

It is an object of the present invention to provide an improved waste oil reclamation method.

5 The present invention provides an improved method of reclaiming waste mineral oil by alkali treatment, removal of water and of highly volatile components, separation of a tar fraction and distillation of the purified oil, wherein the improvement comprises, in combination, the steps of:

10 treating the waste oil with an alkali compound selected from alkali metal compounds and alkaline earth compounds, after the removal of water and highly volatile components and before separation of the tar fraction, and separating the tar fraction during distillation of the oil.

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1 By means of the subject invention's procedural
2 sequence, that is, due to the alkali treatment of the
3 waste oil that already has been dried and liberated of
4 highly volatile particles, the stable emulsion, that
5 also contains solved and and/or emulsified contaminations,
6 is converted into an instable state, so that the sus-
7 pended solids become completely separated during the
8 distillation process of the purified oil, whereby,
9 accordingly, a single step suffices for a complete separ-
10 ation of the suspended solids and for the realization
11 of a flawless purified oil.

12 It is assumed that the alkali treatment, added for
13 the purpose of neutralization of the sour or acidic
14 components and possibly for the saponification of
15 saponifiable contaminations, simultaneously effects an
16 alteration in the interfacing area or boundary surface
17 of the suspended particles due to the previous removal
18 of the volatile parts which were effective as some form
19 of protective colloid. This alteration permits the
20 separation of the suspended particles and the simultaneous
21 distilling off of the suspension medium oil that has
22 already been completely liberated of solid particles,
23 whereby the released, for example saponified, contaminations
24 remain in the tar fraction. In other words, the alkali
25 treatment that follows the removal of water and volatile
26 particles according to the invention, allows the separ-
27 ation of solid and solved contaminations and the yield
28 of a flawless oil in a single distillation step without
29 a separate destruction of the suspension preceding the
30 final distillation.

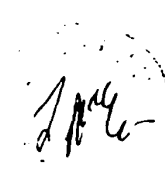
1 The alkali treatment may generally be carried out
2 with aqueous, preferably dissolved alkali at a tempera-
3 ture of 180 to 300° C, preferably at 230 to 260° C, in a
4 closed converter. Higher treatment temperatures within
5 the described range generally result in a lower acid
6 value number and a lower chloride content of the purified
7 oil. The alkali treatment preferably is performed with a
8 water soluble alkali metal compound selected from the
9 group of water soluble alkali metal hydroxides, bicarbon-
10 ates, carbonates, and alcoholates. However, non-soluble
11 or low-soluble compounds of alkaline earth may also be
12 used within the scope of the subject invention. In addi-
13 tion to or instead of sodium, lithium (Li) or potassium
14 (K) may be employed in the alkalies or hydroxides used
15 for waste oil treatment according to the subject invention.

16 The alkali treatment according to the subject invention
17 can also take place simultaneously with the gas oil dis-
18 tillation, e.g. by means of alkali additions before the
19 circulation pump of a forced-circulation evaporator for
20 the gas oil distillation. A gas oil fraction may be dis-
21 tilled off simultaneously during the alkali treatment.

22 After the alkali treatment the waste oil is pre-
23 ferably fed to a gas oil distillation stage and then
24 generally reaches via relief or expansion valve an eva-
25 porator, which preferably is a thin film evaporator,
26 such as a down-flow or up-flow film evaporator, working
27 in cocurrent or countercurrent mode, in which a pitch-like
28 or tar-like residue, containing the particles suspended
29 in the waste oil and further contaminations that are en-
30 trained by these particles and/or distillable impurities,
31 is being extracted. This residue can be used as a tar
32 substitute, such as in street construction.

1 ~~Packably~~, the vapor containing the pure oil vapors and the
2 highly volatile components is fed to a liquid separator
3 in order to separate possible entrained liquid parts
4 and for simultaneous treatment with stripping or scrub-
5 bing steam or water in the liquid phase which evaporates
6 immediately at the given temperatures. The steam treat-
7 ment not only improves the color of the regenerated oil
8 and but also removes substances that are a nasal nuisance.
9 Interestingly, the phosphorus content of the purified
10 oil is also diminished after the steam treatment. It
11 is assumed, that this unexpected result of dry steam
12 application is made possible by the preceding alkali
13 treatment. Besides the known methods of liquid
14 separation, a part of the vapor conduit designed accord-
15 ingly may take over this function.

16 The pure oil vapors are preferably gradually
17 distilled after the steam treatment, wherein the highest
18 boiling fraction is the first fraction obtained and
19 a lower boiling fraction than the next preceding fraction
20 is contained in every further step. The gradual con-
21 densation represents a reversal of the fractional dis-
22 tillation where the separation of the fractions takes
23 place by means of several consecutive condensation
24 levels instead of several consecutive evaporation levels.
25 Fractionalized condensation is advantageous in comparison
26 to fractionalized distillation, in that it is not only
27 more energy conserving, but has furthermore the great
28 advantage, when performed at higher temperatures, that
29 lower boiling contaminations are not condensating with
30 the low boiling oil fraction and are thus not contami-
31 nating the latter, but that they can be trapped separately
32 by means of a cold trap subsequent to the condensation
33 of the lower boiling oil fractions.



1 BRIEF DESCRIPTION OF THE DRAWINGS

2 The subject invention and its various aspects and
3 objects will become more readily apparent from the following
4 detailed description of preferred embodiments thereof,
5 illustrated by way of example in the accompanying drawing
6 which is a diagram of a waste oil reclamation system for
7 carrying out methods according to the subject invention
8 and preferred embodiments thereof.

9 DESCRIPTION OF PREFERRED EMBODIMENTS

10 Homogenized waste oil liberated of coarse contami-
11 nations is fed to the illustrated waste reclamation
12 system 12 through an inlet 13 connected to a pump 14 which
13 applies the waste oil to a circulation evaporator 15.
14 Unless otherwise mentioned herein, the components of the
15 system 12 may be of conventional types as such.

16 The circulation evaporator 15 liberates the waste
17 oil of water and highly volatile components, such as
18 gasoline and solvents. The circulation evaporator 15
19 works at normal pressure or slight vacuum and the product
20 temperature at the evaporator outlet 16 is from 140 to
21 180° C, which corresponds to a residual water content of
22 <0.1%. The latter is advantageous for the subsequent
23 alkali treatment, because less foam formation was observed
24 with lower water content.

25 The vapors issuing from the evaporator outlet 16
26 are applied via a steam-out vessel or flash box 17,
27 wherein evaporation takes place under expansion, to a
28 fractionating column 18 where the vapors are separated
29 into fractions of different boiling ranges. After pass-
30 ing a condenser 19, the distillate 20 can be separated
31 in a decanter into a heavy aqueous and a light organic
32 phase.

1 A pump 21 recirculates waste oil from the flash
2 box 17 back to the first circulation evaporator 15,
3 while a pump 22 feeds waste oil liberated of water and
4 highly volatile components to a heated reactor 23 where
5 it is mixed at a temperature of from 180 to 300° C,
6 preferably from 230 to 260° C, with alkali, such as
7 NaOH, provided by a pump 24 from a storage tank 25. Depend-
8 ing on the quality of the waste oil and with reference
9 to the above disclosure, from 0.2 to 5% NaOH (dry) are
10 used. The waste oil can be heated to the reaction
11 temperature in a series-connected heat exchanger by
12 means of waste oil that has already been treated or
13 then directly in the reactor 23 itself. The residence
14 time in the reactor 23 is approximately 1 to 5 minutes.

15 The waste oil treated with alkali goes from the
16 reactor 23 to an additional circulation evaporator 26,
17 where a gas oil fraction 27 is distilled off at an
18 average vacuum of 5000 to 50000 Pa at approximately
19 330° C. In this respect, the abbreviation "Pa" stands
20 for Pascal, which is the main pressure unit comprised
21 in the International Unit System (S.I. System) and
22 defined as the force of one Newton per square meter.
23 The above mentioned Tkac et al US Patent 4,333,822 has
24 in col. 4 a table of equivalents of Pascal in other
25 measurement systems.

26 It is advantageous to attain the temperature re-
27 quired for this distillation by means of cooling after
28 the alkali treatment and heating in the circulation
29 evaporator 26. A heat exchanger used for the cooling
30 process can simultaneously be employed for the pre-heating
31 of the used oil preceding the alkali treatment.

1 A steam-out vessel or flash box 28 applies output
2 waste oil of the evaporator 26 to a second fractionating
3 column 30 which with the aid of a condenser 31 effects
4 separation of a gas oil fraction 27 with a desired flaming
5 point and with a desired viscosity.

6 A pump 32 recirculates waste oil to the circulation
7 evaporator 26, while a pump 33 applies the waste oil
8 liberated of gas oil from a second flash box 28 via a
9 liquid separator 34 to a thin-film or thin-layer evapor-
10 ator 35, where it is subjected to a total distillation
11 under vacuum. The thin-layer evaporator 35 can be operated
12 statically or mechanically and it can be either a down-
13 flow or an up-flow film evaporator. The vacuum distillation,
14 carried out here in the counter-current mode at a heating
15 temperature of from 350 to 380° at 100 to 500 Pa, effects
16 a separation of the waste oil suspension into a bottom
17 product or tar fraction 36 and a vaporous lubrication
18 oil fraction. The bottom product 36 is drawn off by a
19 pump 37 at approximately 300° C and can be used as a
20 tar substitute.

21 Vapors from the vapor head 39 reach the liquid
22 separator 34 at a temperature of approximately 270° C and
23 entrained droplets are separated in the evaporator 34
24 and fed again to thin-layer evaporator 35 via a line 42.

25 In the illustrated embodiment, the waste oil sus-
26 pension is fed from the gas oil distillation into the
27 liquid separator 34 from where it is led together with the
28 separated liquid into the thin-layer evaporator 35. This
29 method has the advantage that highly volatile components
30 can already evaporate in the liquid separator 34. However,
31 the waste oil suspension alternatively can be fed directly
32 into the thin-layer evaporator 35, such as by means of
33 pump 33.

1 In the liquid separator 34, the vapors are preferably
2 subjected to a scrubbing or strip steam treatment, as
3 indicated by a water or steam supply line 44 to or in the
4 separator, whereby color and olifactory quality of the
5 resulting condensate can be improved and its phosphorus
6 content diminished.

7 The vapors liberated from liquid parts subsequently
8 reach a first direct-contact or mixing condenser 46, where
9 they are condensated at a heat exchanger having a con-
10 densate circulating therein at a temperature that is
11 approximately 10° C below the boiling point of the fraction
12 to be obtained. The first heavy oil fraction 47 may thus
13 be separated and drawn off with a pump 48.

14 An additional direct-contact or mixing condenser 49
15 is operated in the same manner but at a lower temperature
16 and provides a second lighter oil fraction 50 which may
17 be drawn off with a pump 51.

18 Due to the strong turbulence in the mixing condensers
19 46 and 49, high efficiency is achieved and a frequently
20 observed demulsification and deposits on cooling surfaces
21 of other condensers is thus avoided.

22 The vapors leaving the second condenser 49 still have
23 a temperature of approximately 200° C and contain small
24 amounts of highly volatile substances that can be trapped
25 by a cold trap 52 in order to avoid that any contami-
26 nations reach the vacuum pump. The resulting condensate
27 appears at the systems output 53.

28 Due to the application of a two-stage direct-contact
29 or mixing condenser system, whose stages operate just
30 slightly below the boiling point of the particular fraction,
31 the quantity ratio of the two oil fractions as well as
32 their boiling point can be varied within larger limits,
33 whereby those two values can be adjusted practically almost
34 independently from each other.

EXAMPLE

1 The following Table presents the results of the
2 analyses of oil reclaimed according to the method of the
3 subject invention, as disclosed above and illustrated. As
4 may be seen from these listed results and comparative
5 analyses, the subject invention and its preferred
6 embodiments obtain a flawless fraction of gas oil and
7 a light and a heavy lubricating oil fraction in a
8 minimum amount of processing steps. Relatively inexpensive
9 NaOH may be employed for the alkali treatment as disclosed
10 above.

11 According to the method subject of the invention, the
12 minimization of the processing steps, particularly the
13 reduction in distillations, diminishes the danger of thermal
14 cracking and the reduction in yield caused thereby. Accord-
15 ingly, the yield of the method and apparatus of the subject
16 invention is at least 90% of the distillable portion of the
17 waste oil, which increases the economy of operation sub-
18 stantially over those achieved by the prior art.

19 Reverting now to the following Table, the composition
20 of the waste oil used in the tests recorded therein is
21 listed in column 0. The alkali compound used for treatment
22 in these tests was NaOH present in an amount of 0.7% by
23 weight of solids, and ASTM and DIN-NMF testing conditions
24 were observed.

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TABLE

Analysis	0	1	2	3	4	5	6	7	8	9	10	11
Color (ASTM D 1500)	>10	2	1.5	1.5	6.5	1.0	4.5	2.0	6	4	2	--
Flash point (ASTM D 92/93) °C	183	110	108	167	218	--	193	226	236	--	223	--
Viscosity (ASTM D 445)												
40°C mm ² /s	52.1	*13.71	*9.97	13.33	32.6	15.2	24.1	30.7	52.6	82.4	33.1	--
100°C mm ² /s	8.65	--	--	3.09	5.39	3.36	4.49	5.2	7.32	9.69	5.49	--
Viscosity index (ASTM D 2270)	143	--	--	84	98	87	95	98	98	95	101	--
Total Acid Number (ASTM D 664)	1.52	--	--	0.03	0.69	0.04	0.06	0.03	0.12	0.01	0.02	--
mgKOH/g												
Total Base Number (ASTM D 2896)	2.98	--	--	0.21	0.24	0.19	0.41	0.25	0.26	0.61	0.23	--
mgKOH/g												
Oxide ash content (ASTM D 482)	0.66	<0.001	<0.001	0	0	0	0	0	0	0	0	8
g/100g												
Pour Point (ASTM D 97) °C	-33	--	--	--	-6	-18	-6	-3	-6	-6	-6	--
Density at 15°C kg/m ³	0.888	865	857	--	877	868	873	875	880	885	875	--
Ageing characterist.-- (DIN 51 352) g/100g	--	--	--	--	0.53	--	--	0.43	0.58	--	0.27	--

FIRST CONTINUATION OF TABLE

Analysis	0	1	2	3	4	5	6	7	8	9	10	11
Sulphur content g/100g	0.72	0.65	0.45	0.42	0.53	0.42	--	0.48	0.58	0.61	0.48	1.65
Chlorine content g/100g	0.12	0.1137	0.0468	0.045	0.033	0.007	0.01	0.01	0.017	nn	nn	0.3
Silver content mg/kg	nn	nn	nn	nn	nn	nn	nn	nn	nn	"	"	nn
Aluminum content mg/kg	16	"	"	"	"	"	"	"	"	"	"	70
Boron content mg/kg	23	~12	"	"	2	"	"	"	"	"	"	66
Barium content mg/kg	130	nn	"	"	nn	"	0.5	"	"	"	"	440
Calcium cont. mg/kg	1200	"	0.4	"	"	"	0.4	"	"	0.1	"	4600
Cadmium cont. mg/kg	3.1	"	nn	"	"	"	nn	"	"	nn	"	8.8
Chromium cont. mg/kg	4.2	"	"	"	"	"	"	"	"	"	"	17
Copper content mg/kg	32	"	"	"	"	"	"	"	"	"	"	94
Iron content mg/kg	160	"	"	"	"	"	1.8	"	"	"	"	540

SECOND CONTINUATION OF TABLE

Analysis	0	1	2	3	4	5	6	7	8	9	10	11
Magnesium cont. mg/kg	170	nn	nn	nn	nn	nn	nn	nn	nn	nn	nn	460
Manganese cont. mg/kg	3.4	"	"	"	"	"	"	"	"	"	"	10
Molybdenum cont. mg/kg	2.1	"	"	"	"	"	"	"	"	"	"	7.2
Nickel content mg/kg	2.8	"	"	"	"	"	"	"	"	"	"	8.6
Phosphorus cont. mg/kg	730	230	27	22	100	5.5	7.5	6.4	71	"	3.6	2200
Lead content mg/kg	960	nn	0.8	nn	nn	nn	nn	nn	nn	"	nn	3000
Silicon content mg/kg	32	11	nn	1.5	4.7	0.7	"	1.1	4.5	"	"	162
Tin content mg/kg	3.9	nn	"	nn	nn	nn	"	nn	nn	"	"	15
Titanium content mg/kg	3.9	"	"	"	"	"	"	"	"	"	"	20
Vanadium content mg/kg	0.4	"	"	"	"	"	"	"	"	"	"	2.8
Zinc content mg/kg	510	"	"	"	"	"	0.9	"	"	"	"	3000

10

10

The individual probe numbers in the Table represent:

- 0 feed
- 1 gas oil, without alkali treatment, cold condensation,
pilot plant
- 2 gas oil, with alkali treatment, hot condensation, pilot plant
- 3 gas oil, with alkali treatment, hot condensation, steam
treated, pilot plant
- 4 light oil fraction, without alkali treatment, pilot plant
- 5 light oil fraction, with alkali treatment, glass evaporator
- 6 light oil fraction, with alkali treatment, pilot plant
- 7 light oil fraction, with alkali treatment, steam treated,
pilot plant
- 8 heavy oil fraction, without alkali treatment, pilot plant
- 9 heavy oil fraction, with alkali treatment, glass evaporator
- 10 heavy oil fraction, with alkali treatment, pilot plant
- 11 tar fraction, with alkali treatment, pilot plant

Meaning of the symbols in the Table:

- * viscosity measured at 20°C
- not known
- nn not detectable

The claims defining the invention are as follows:

1. An improved method of reclaiming waste mineral oil by alkali treatment, removal of water and of highly volatile components, separation of a tar fraction and distillation of the purified oil, wherein the improvement comprises, in combination, the steps of:

treating the waste oil with an alkali compound selected from alkali metal compounds and alkaline earth compounds, after the removal of water and highly volatile components and before separation of the tar fraction, and separating the tar fraction during distillation of the oil.

2. The method according to Claim 1 wherein said alkali treatment is performed with aqueous, dissolved alkali.

3. The method according to Claim 1 wherein said alkali compound is selected from the group of alkali metal hydroxide, bicarbonate, carbonate and alcoholate.

4. The method according to Claim 1, 2 or 3 wherein said alkali treatment is performed at a temperature of from 180 to 300°C.

5. The method according to Claim 4 wherein said temperature is 230 to 260°C.

6. The method according to any one of Claims 1 to 5 including the step of distilling off a gas oil fraction simultaneously during the alkali treatment.

7. The method according to any one of Claims 1 to 5 including the step of distilling off a gas oil fraction subsequent to the alkali treatment.

8. The method according to any one of Claims 1 to 7 including the step of separating the tar fraction and distilling the oil in a thin layer and under vacuum.

9. The method according to any one of Claims 1 to 8 including the step of treating oil vapours developing during distillation of the oil with water vapour.

10. The method according to any one of Claims 1 to 9 including the step of separating liquid components entrained during the distillation of the oil before a condensation process.

11. The method according to any one of Claims 1 to 9 including the step of condensing the oil in two steps during distillation.

12. The method according to any one of Claims 1 to 11 including the step of condensing the oil in a direct-contact or mixing condensers.

13. The method according to any one of Claims 1 to 12 including the step of condensing vapours of said oil during distillation of the oil at a

1112

temperature of approximately 10°C below the boiling temperature of the fraction to be obtained.

14. A method of reclaiming waste mineral oil substantially as hereinbefore described with reference to the Example and/or accompanying drawing.

15. The product of the method of any one of Claims 1 to 14.

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BUSS AG

Patent Attorneys for the Applicant

SPRUSON & FERGUSON.

J. B. C.

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